Standard Operating Procedure for the Digestion Preparation of Soil, Sediment, and Sludge Samples

1.0 Location

This procedure is performed in the spectroscopy laboratory, room 305.

2.0 Purpose

This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by furnace atomic absorption spectroscopy (GFAA) or by inductively coupled plasma spectroscopy (ICP).

3.0 Scope

This procedure prepares the samples for analysis of analytes by GFAA and ICP as listed below in the first column and by ICP only in the second column.

GFAA and ICP	ICP
Arsenic	Aluminum
Beryllium	Barium
Cadmium	Calcium
Chromium	Copper
Cobalt	Magnesium
Iron	Manganese
Lead	Nickel
Molybdenum	Osmium
Selenium	Potassium
Thallium	Silver
Vanadium	Sodium
	Zinc

4.0 Reference

This method is referenced in SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Laboratory Manual Volume 1A. Method 3050A.

5.0 Sample Handling and Preservation

Samples should be collected following the procedures for metals listed in the EPA document SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Laboratory Manual Volume 1A, Method 3050A Section 6.0.

6.0 Apparatus and Materials

6.1 Glass or plastic 50 ml tube

6.2 Filter Paper -- Whatman No. 41 or equivalent.

6.3 Analytical Balance -- Mettler AE160 or equivalent.

6.4 Ribbed watch glasses or equivalent

6.5 Electric Hot Plate. Capable of maintaining a temperature of 90-95^{o} C.

6.6 Sample digestion reagents including

6.8.1 Reagent Water -- ASTM Type II water or better

6.8.2 Nitric Acid -- Instra-analyzed

6.8.3 Hydrochloric Acid -- Instra-analyzed

6.8.4 Hydrogen Peroxide (30%) -- High Purity

7.0 Procedures

7.1 Weigh out a representative 3.0 g sub-sample (talk to chemist in charge to see if a different amount is needed) into a 50 mL tube. Record the weight to the nearest 1 mg. For samples with low percent solids a larger sample size may be used as long as digestion is completed.

7.2 Add 10 mL of 1:1 nitric acid to the beaker, mix the slurry.(5:5) Heat the sample to 95^{o} C, cover with a ribbed watch glass, and reflux for 10 to 15 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated nitric acid and reflux for 30 minutes. Repeat this last step to ensure complete oxidation. Remove the ribbed watch glass and allow the solution to evaporate to 5 mL without boiling .

7.3 After step 7.2 has been completed and the sample has cooled, add 2 mL of water and 2 mL of 30% H_2O_2. Return the tube to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the sample tube.

7.4 Continue to add 30% H_2O_2 in 2 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.(Do not use more than 10 ml)

7.5 Heat the sample until the volume has been reduced to approximately 5 mL. After cooling, filter the sample through Whatman 41 filter paper or equivalent. Dilute to 100 mL and pour in a 250 mL bottle.

7.7 Prepare duplicates, blanks, and spikes as in 7.1 through 7.6. To the spike, add 10 uL of 1000 ug/mL stock standard solution of the applicable metal.

8.0 Quality Assurance/Quality Control

8.1 For each batch of samples processed, a preparation blank should be carried throughout the entire sample preparation and analytical process. This blank will be useful in determining if samples are being contaminated.

8.2 Duplicate samples and spiked samples should be included at a 10% frequency with at least one of each per batch of samples.

9.0 Data Analysis

The result in ug/g should be calculated as follows:

Result = (A X B / C) X 1000 where

A = Result from analysis in ug/L
B = Volume to which the digested samples were brought in L (0.100 L)
C = Weight of the sample digested in g

10.0 Documentation

Record all weights and keep all records of analysis. Enter the calculated value into the lims via a worklist and record the value onto the bench sheet. Keep all other relevant information with the worklist and place it into the individual metal's parameter log book.

11.0 Records

All recorded information shall be maintained and kept in the relevant metal's log books.